UNICAT Newsbriefs

Issue 28
March 11, 1999

Edited by R. Simpson and H. Chen

** UNICAT Staff Updates **

• *Pete is married*
Dr. Peter Jemian and Dr. Alta Mekaelian were united in marriage at St. Paul's Armenian Apostolic Church in Waukegan, IL on February 13, 1999. They recently returned from a honeymoon in the Lake Louise area of the Canadian Rockies. Dr. Mekaelian is a dentist in practice with her brother. Pete and Alta met when they were participants in a musical group entertaining the residents of a retirement home. They reside in Lake Bluff, IL, a 10 minute drive for Alta and an hour drive for Pete. He wasn't at all concerned about the travel distance and said you could see the roller coasters at Great America from their home. Congratulations to Pete and Alta, we wish you many happy years together!

• *News from David Robinson*
We recently learned that David became a husband and a father during 1998. His son George was born in late fall. In a recent message he said his lifestyle has certainly changed since leaving UNICAT both in regard to his personal and professional life. David can be reached at david.robinson@oxinst.co.uk

• *Jan Ilavsky is now at APS*
UNICAT is happy to extend a warm welcome to Jan Ilavsky of NIST who has now relocated to the APS. Jan will be helping further the NIST research mission at the APS, and will help build and maintain the UNICAT facility. On your next visit to the APS, please be sure to stop by and introduce yourself to Jan. He can be contacted by e-mail at ilavsky@aps.anl.gov, and by phone at 630-252-0866.

** News from APS **

• *David Moncton Leads the Spallation Neutron Source Project*
The following is an excerpt from a received from Susan Strasser and quoted from David Moncton:
"I have recently been asked to take leadership of the $1.36B Spallation Neutron Source Project at Oak Ridge National Laboratory. This project is a collaboration among five National Laboratories including Argonne, and it is of very high national priority. Under the terms of the agreement I have made with the directors of both ANL and ORNL, I will spend the majority of my time over a minimum of the next two years on this assignment, but I will remain an Argonne/APS employee. I will retain the position of Associate Laboratory Director over this period, but most of my APS management responsibilities will be delegated to John Galayda and Gopal Shenoy. I am very confident that the APS will continue to thrive under their leadership and look forward to returning full time to APS in the future. I will be doing

this job on a commuting basis, so I do plan to be at the APS frequently and to keep in close touch with activities here. I have great confidence that the APS will continue its record of excellence."

** IBHE-HECA XCITE News **

XCITE is a collaborative group supported by an IBHE-HECA grant from the State of Illinois. It involves 6 universities in the state: UIUC, NWU, U. Chicago, IIT, Northern IL, and Southern IL.

• ***Williams joins XCITE as Outreach Coordinator***

On January 4, 1999, David Williams became the Coordinator of Educational Outreach for the X-ray Collaboration for Illinois Technology and Education (XCITE).

David brings an exceptional breadth of experience to XCITE educational outreach. Prior to accepting this position, he was the Curriculum Coordinator for the Manufacturing Technology Program at Illinois Institute of Technology (IIT). He has worked as an independent contractor and consultant in higher education, workforce development, association management, and public affairs, as well as in adjunct faculty assignments. Before his association with IIT, he was the Director of Institutional Planning for the City Colleges of Chicago.

You may have met David in his travels to become acquainted with the HECA University grant partners. He is also meeting with the XCITE Executive Committee members and other administrators and faculty on those campuses. He has met with representatives of COM-CAT, Harold Myron (ANL-DEP) and Susan Barr-Strasser in the APS User Office. He has established contacts with community colleges throughout the state and has secured a spot on the agenda of the business meeting for the Illinois Community College Economic/ Workforce Development Association on April 15.

For the remainder of this fiscal year, David will be occupying space in Argonne Building 223, Division of Education Programs offices. When the Sector 34 LOM is ready for occupancy, he will relocate.

David can be reached by e-mail at d-williams7@nwu.edu.

• ***Highlights from XCITE (X-ray Collaboration for Illinois Technology and Education) Executive Committee Meeting.***

(The following information was taken from minutes of the meeting).

The XCITE Executive Committee met at the Advanced Photon Source (APS), Argonne National Laboratory on February 18. In attendance were Lydia Villa-Komaroff, (Northwestern University), Haydn Chen (University of Illinois at Urbana-Champaign), Clyde Kimball (Northern Illinois University), Keith Moffat (The University of Chicago), Timothy Morrison (Illinois Institute of Technology), John Quintana (Northwestern University), Joy Talsma (The University of Chicago), David Williams (XCITE Coordinator of Educational Outreach), Dale Wittmer (Southern Illinois University at Carbondale), and Paul Zschack (University of Illinois at Urbana-Champaign).

Location of XCITE outreach office: Temporarily the office will be located in the Division of Educational Programs, Building 223, ANL. As soon as the sector 34 LOM is available the office will be moved to that location and include space for a reception area for APS visitors. In this location, the coordinator will be in an environment where his primary activities are centered. It is important to keep the XCITE outreach separate from the Argonne outreach umbrella however Harold Myron should feel involved with XCITE's outreach activities.

Although there is concern for industrial outreach the committee feels the primary audience for XCITE outreach efforts should be higher education and it was agreed that XCITE outreach activities must be visible.

There was agreement that it is important to make it clear that XCITE works with Argonne, COM-CAT, and Harold Myron, and not for Argonne, COM-CAT, or Harold Myron.

Harold Myron distributed announcements of the "first United States National School on Neutron and X-ray Scattering to be held at Argonne National Laboratory, August 16-27, 1999." Graduate students attending U.S. universities and majoring in physics, chemistry, or materials science are encouraged to apply. A limited number of postdoctoral appointees and junior scientists from universities, national and industrial laboratories may also be selected to attend the School, as allowed by class size. Myron expects 48 students for the School, which is being funded by $100,000 from the Department of Energy.

Consensus was reached that the XCITE outreach project for this fiscal year would be a two-day workshop. This effort should be a "pilot" for a proposal for a more ambitious and broader—reaching "school." The two-day workshop will be in July or August 1999. Morrison will be chair of the program committee. The Executive Committee will be developing the scientific program. Williams will chair the workshop. Information on the workshop will be forthcoming.

<u>APS-PEB Review: Sector 34-ID</u>

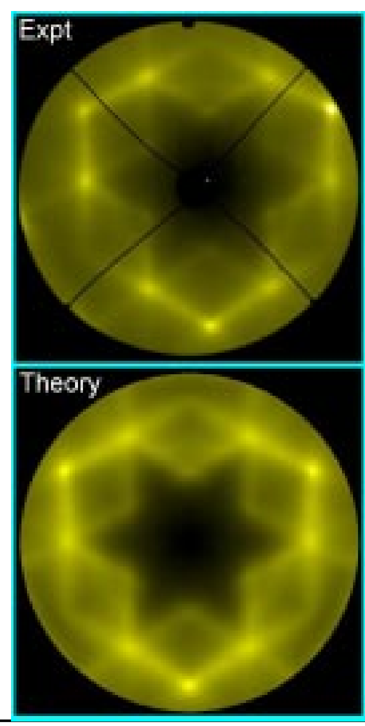
Ian Robinson, Paul Zschack, Curtis Benson and Haydn Chen attended the annual APS-PEB review for the construction of the 34-ID beam line. The review was held at APS/ANL, on Feb. 18, 1999. Chen made a brief report on the scientific scope of the dedicated microbeam facility, reviewed the funding profile and past accomplishments. Robinson discussed design details, cost estimates and project timelines.

** <u>Highlights of Scientific Results</u> **

- ***Thermal Diffuse Scattering Patterns:***

The first publication based upon the use of UNICAT Sector 33-ID beamline appeared in Physical Review, **B59**, 3283 (1999). The title of this work is "Pattern of X-ray Scattering by Thermal Phonons in Si", which is coauthored by Z. Wu, H. Hong, R. Aburano, P. Zschack, P. Jemian, J. Tischler, Haydn Chen, D.A. Luh and T.C. Chiang.

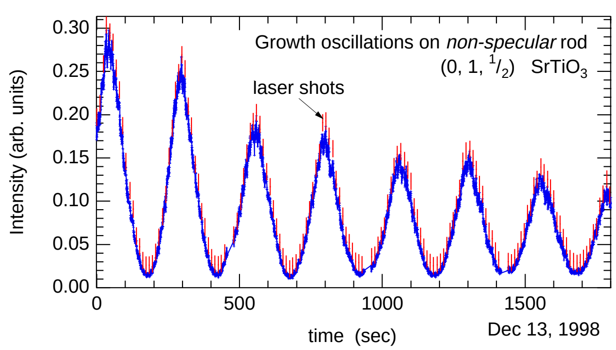
A short summary of this work is given below: Intensity distribution of x-ray scattering by thermal phonons in Si was recorded using



synchrotron radiation from the 7 GeV storage ring at the Advanced Photon Source. A high-energy beam sent through a Si (111) wafer in a transmission Laue geometry yielded a threefold symmetric pattern for the scattering cross section with rich details governed by the phonon dispersion, population, and polarization. Theoretical patterns were shown to have good agreement with experimental results. The present experiment is a demonstration of a new approach for phonon studies. The pattern displayed on a logarithmic scale, show intricate features that are sensitive to details of the phonon properties. This approach (and its extension to include temperature-dependent measurements) should be useful for investigations of a variety of materials. Phase transitions involving phonons are an area of interest.

- ***Real-Time X-ray diffraction:***

Tischler, Yoon, Larson, Eres and Lowndes of ORNL group have studied time-resolved x-ray diffraction studies for ZnO grown on c-plane sapphire and for homoepitaxy on $SrTiO_3$ during laser ablation deposition. To our knowledge, these are the first experiments. Some conclusions are:



- deposition and growth time-scales significantly longer than seconds have been observed;
- incommensurate ZnO islanding is observed after single layer deposition at 400 and 585° C; and
- ZnO x-ray coherence lengths smaller than AFM cluster sizes for growth temperatures of 400 and 585° C.

This work was presented in the 1998 Fall MRS meeting in Boston.

** Commissioning Experiments **

In late August 1997 sector 33ID entered into the commissioning phase. The following commissioning experiments have been carried out by UNICAT members and their associates.

Surface Reconstruction / Interface Structures
T-C. Chiang, H. Hong, Z. Wu & P. Zschack

Diffuse Scattering in $Ag_{50}Pd_{50}$ Solid Solution
C.J. Sparks, G.E. Ice, W. Schweika & P. Zschack

Phonon Imaging in Si
Z. Wu, H. Hong, R. Aburano, P. Zschack, P. Jemian, J. Tischler, H. Chen, D-A. Luh, & T-C. Chiang

Induced Magnetic Moment in Co/Ir Thin-Film Multilayers
M. Salamon & K. O'Donovan

Use of X-Ray Microbeams for Cross-Section Depth-Profiling of MeV Ion-Implantation Induced Defect Clusters in Si.
M. Yoon, B. Larson, J. Tischler, T.E Haynes, J-S. Chung, G.E. Ice & P. Zschack

Zeolite & Metal Phosphate Structures - Powder Methods
R. Broach & R. Kirchner

Thin-Film Domain Structures using CXRS
I.K. Robinson, J. Pitney & D. Fanning

USAXS Commissioning
Andrew Allen, Pete Jemian, Gabrielle Long & Jan Ilavsky

Surface Diffractometer Commissioning
H. Hong, Z. Wu & P. Zschack

Pulsed Laser Ablation Growth (Chamber Commissioning)
J. Tischler, B. Larson, M. Yoon, J. Budai, G. Eyres, & D. Lowndes

EXAFS Studies of Catalysts
Simon Bare, George Mickelson & Frank Modica

Anomalous Scattering from Inert Gases
P. Zschack

Surface Reconstruction / Interface Structures
T-C. Chiang, H. Hong, Z. Wu, H. Chen & P. Zschack

Phase Transitions
T-C. Chiang, Z. Wu, H. Hong, P. Zschack & H. Chen

USAXS Commissioning
A. Allen, P. Jemian, G. Long & J. Ilavsky

Surface Reconstruction / Interface Structures
T-C. Chiang, H. Hong, Z. Wu, H. Chen & P. Zschack

Thermal Diffuse Scattering Imaging
Z. Wu, H. Hong, P. Zschack, H. Chen & T-C. Chiang

Inelastic X-Ray Scattering from Insulating Cuprates
Peter Abbamonte, Miles Klein, Eric Isaacs, Clem Burns & Paul Zschack

Laser Ablation Growth of ZnO Thin Films
J. Tischler, B. Larson, M. Yoon, J. Budai, G. Eyres, N. Tamura & D. Lowndes

Phonon Dispersion Determination with Thermal Diffuse Scattering Imaging
Z. Wu, M. Holt, H. Hong, P. Zschack, P. Jemian, J. Tischler, H. Chen & T-C. Chiang

RSXD from Strained Layers
H. Hong, Z. Wu, P. Zschack, T-C. Chiang & H. Chen

High Resolution Powder Diffraction of Zeolites
R. Broach

Laser Ablation Growth of $SrTiO_3$ Thin Films
J. Tischler, B. Larson, M. Yoon, J. Budai, G. Eyres & D. Lowndes

USAXS Characterization of Steel Alloys
P. Jemian & D. Alexander

Surface Coherent Diffraction
I.K Robinson, J. Pitney & C. Benson

USAXS of Ceramic Colloidal Suspensions
Andrew Allen, Pete Jemian, Gabrielle Long & Jan Ilavsky

USAXS Measurments of Zeolite Crystallization
R. Broach, L. Patton, P. Jemian, R. Willis & H. Chen

Thermal Diffuse Scattering from Relaxor Ferroelectrics
A. Tkachuk, Z. Wu, H. Chen, P. Zschack & H. You.

Real-time Observation of Surface Structural Transformations
H. Hong, Z. Wu, M. Holtz, P. Zschack, T-C. Chiang & H. Chen

Laser Ablation Growth of $SrTiO_3$ Films
J. Tischler, B. Larson, M. Yoon, G. Eyres, & C. Rouleau

Spacer-layer Magnetization in Magnetic Superlattices
M. Salamon, K. O'Donovan, T. Park & H. Yanagihara

** Sector 33 Updates **

33ID Commissioning activities continue on our insertion device beamline. In each running period and shutdown, we are able to identify areas for improvement to the optical performance of the beamline. Much of this work has been gaining control over the thermal issues due to Compton scattering onto the second crystal focusing mechanism. For example, with the help of the XFD water group, we have added filters and Cu mesh to the DI water lines running to the second crystal and Compton shield. The reduced vibrations were immediately noticeable.

The UNICAT displex that is compatible with the Huber 4-circle diffractometer has arrived and is functioning well. The first experiment with this cryostat demonstrated reliable control, very fast cool-down, and a base temperature better than 6K! We are now fabricating a vacuum shroud to accommodate our Be hemisphere to permit a wide access to reciprocal space.

During the next running period, we will use the Newport goniometer for the first time. Although the axes can be moved, and control by SPEC is available, we have much to learn about this kappa geometry and servo motor controlled goniometer. I know expectations are high that this new gonimeter will be a tremendous benefit to many of our scattering programs.

We have learned that the coatings on our ID mirrors are not quite what the mirror vendor proposed. Although the focusing is uneffected, the consequence of this is that good harmonic rejection is acheived only for energies above about 9-10 KeV. We are now investigating options to improve this performance. For the short term, one solution is to mount a smaller float-glass mirror on the beam conditioning table. Discussions with the mirror vendor regarding a long term solution are underway.

33BM Planning and procurement activities continue toward the development of our Bending Magnet Beamline. The DCM final design review was held in December, and the final design has been accepted. The current expectation is for delivery of the DCM in January, 2000. Other components for the BM beamline are in various stages of procurement. For example, we have already received our white-beam slit and bremsstrahling collimator. Other long-lead time items such as the photon shutter and mirrors are in fabrication. Preliminary results for figure error and roughness from the monochromatic focussing mirror are extremely encouraging!

LOM The construction and buildout of offices in the 438-E pentagon has begun. We expect to have beneficial occupancy of 11 new offices by mid-April. This new space is necessary with the addition of new resident scientists and students.

** New Listserv update **

Beginning Apr. 1 the listserv at MRL will be the only listserv for UNICAT. The list at ORNL has been moved to the new service and Jon Tishler has been asked to deactivate the ORNL list on Mar. 31. You should have received a message confirming your inclusion in the list. To use the list you should send messages to:
UNICAT@Ginny.mrl.uiuc.edu

** REMINDER **
Submission of Research Performed on UNICAT facility

Following is a list of the required information to be provided for publications resulting from research done using the UNICAT beamlines. <u>All materials shall be mailed to the UNICAT Headquarters Office as follows. Ramona Simpson will be the point of contact.</u>

Mrs. Ramona Simpson
UNICAT Administration Office
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University of Illinois
104 South Goodwin Avenue
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<u>Reprints from Journals, Extended Abstracts or Book Chapters:</u>
2 hard copies of each reprint including the proper acknowledgement (found in the Management Plan and Independent Investigator documents on our web pages) to UNICAT, APS. **<u>The acknowledgement must be as stated for you to receive credit for the paper when reports go to our funding agencies</u>**.

<u>Invited and Contributed Talks:</u>
Title, presenter, conference/symposium name, where given, dates, etc.

<u>Ph.D. Thesis:</u>
1 hard copy of the thesis.

For all papers (reprints, invited talks, etc.): Provide also a digital copy in PDF format for inclusion on our web pages. If you need assistance in creating these files please contact Paul Zschack or Pete Jemian.

** <u>Sector 34 Updates</u> **

(Please direct feedback to Ian Robinson)

The design of the sector 34 ID has reached a mature stage now with most of the components defined and fixed in their positions. Our first major adventures with procurement have now started. The hutch specification was finally approved by APS and is now out for quotation. A considerable effort was spent to put together a package of design drawings of major components - shutters, masks and beam transports - which has been sent out for bid by the University grants and contracts office. This included two of our own designs for special masks needed to divide the beam. We therefore expect to be ordering these soon.

The next round of procurement will be for support tables for all the beamline components. We have found two sources of 'economical' table designs which we are comparing with our needs. In order to specify all the tables, we have to consider all aspects of the survey procedure that will be used to situate the critical components. This will determine how many independently surveyed steps there will be in the final assembly, and hence how many degrees of freedom we need in our design. We have been advised to avoid any requirement of 'shimming' or 'shoving' because of the high cost of surveyors' time!

Meanwhile we are starting to take delivery on the first parts needed to assemble the mirror positioner. Some of these are still weeks away from completion, but we hope to start assembly in the early summer. We had one setback with our order of bearings and slides from Franke in Aalen. Franke does not FOB from their US sales office, which obliges us to import them ourselves. The parts apparently arrived at ORD but became lost before reaching the hands of the University's customs agent. While those parties sort out their differences, we have had to reorder the parts. Susan Johnson has done a heroic detective job of chasing down the story.

In February we made our annual state-of-the-beamline address to the PEB. There was some discussion on the possibility of future operations of 34ID with two undulators, with one supplying each branch. Sector 4, currently under construction, already plans this kind of operation to allow more independence of the two branches. We have reflected on the implications for our design for 34ID. Two *in-line* undulators in the front end would not be very interesting because the spectrum of one branch would be received fully by the other branch; in practice, this might even be worse than the constraints of sharing a single undulator, where at least the spectrum can be trusted to be fixed. As planned at sector 4, two *canted* undulators with a separation of 270µrad (maximum value dictated by the machine operation) would allow the peaked part of their spectra (which change most with gap) to be totally separated. Unfortunately our design cannot accommodate such a large beam separation without serious modification of the Bremsstrahlung shielding, but with one minor change we can accept two beams canted by about 135µrad, giving 3.9mm separation at the beam-splitting mirror. This should be sufficient to reject cross contamination of the peaked portion of the two spectra by about 90%. We are therefore planning to open that single aperture to allow for this future improvement.

The next major component to go out for bid will probably be the special diffractometer for the coherent diffraction experiments. We have thought a lot about how to design this and would like to encourage opinions from the Unicat community before we close it out. Please respond to ikr@uiuc.edu. The design outline is summarized in the next section of the newsletter for your information.

Excerpts from the Design Specification of the CXD Diffractometer

The front experimental hutch (34ID-C) of the APS 34-ID beamline will perform experiments using Coherent X-ray Diffraction (CXD) as its sole function. The unique demands of CXD are that a very small beam (typically 5 microns) be defined a short distance (10 to 20mm) in front of the sample. Diffraction from the sample is angularly resolved by a detector that is typically quite far away, at the end of a detector arm that is up to 3m long. The three roles, i) support of the beam-defining apertureø, ii) oòientationFof the sample in tée beam, and iii) angular positioning of the detector arm, are the primary functions of the CXD diffractometer.

The 34-ID beamline is also special in that it is planned to operate two experiments *simultaneously*. Its undulator beam is divided by a mirror at 30m into two beams that supply independently shuttered hutches. Since the CXD takes place in the first hutch, centered at 51m from the source, it is inconvenienced by the *permanent presence* of a shielded pipe carrying the second beam. The diffractometer must therefore operate in a space that does not encroach that of the pipe, whose outer wall lies just 180mm from the diffractometer’s center. For this reason we will employ the style of the kappa-axis diffractometer for *both* sample and detector motions.

Because of the space restrictions, the diffractometer must be able to provide both vertical and horizontal scattering-plane geometries. In particular, horizontal and vertical detector motions must be available on an equal footing. In the double-kappa configuration, scattering planes *in between* horizontal and vertical are also provided. Finally the detector arm should be rotatable about its own axis: this has several advantages such as facilitating polarization analysis or alignment of exit slits or linear detectors along selected reciprocal-space directions. Since it is impossible to make a fully rotatable arm that is also completely rigid, relaxed design tolerances have been specified.

It is expected that the diffractometer specified in this document can be fabricated from a standard off-the-shelf kappa-goniometer attached to the side or base of an L-shaped support. The most suitable position for the detector arms is on the back of the support. Although these are in a custom arrangement, the specification can be met using off-the-shelf rotary tables. Because their functions are quite separate, it is conceivable that the sample goniometer and the detector-motion axes might be provided by different vendors.

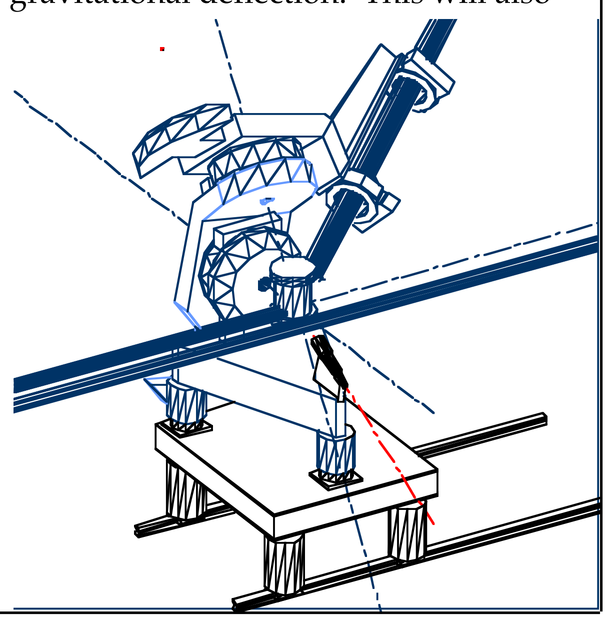
In the CXD hutch is a transport system that allows the diffractometer table to be installed and also moved along the direction of the beam, with a total range of 1.0m travel. This allows the diffractometer to couple to a UHV sample environment in the *front* position or to operate with samples mounted in air or local environments in the *back* position.

There is need for an optical bench opposite the diffractometer, rigidly connected to the support, and a rigid stand for holding input beam components. The input beam stand must disassemble sufficiently to slide under the UHV sample preparation chamber in the front position, for which the available space is limited.

The 'double kappa' instrument has a total of six principal axes, three for the sample and three for the detector, as illustrated in the figure. The three sample axes follow a standard kappa sequence: omega, kappa, phi. The three detector axes follow the same sequence: omega_D, kappa_D, phi_D. The angle of inclination of the kappa axes with respect to both omega and phi, here called 'alpha', must be greater than 45° and is usually chosen to be 50° or 60°, but could adopt a different value if necessary. The angle of inclination of the kappa_D axes with respect to both omega_D and phi_D, here called 'alpha_D', must be precisely 45° because this serves to protect the adjacent white beam transport from infringement. This means that the detector arm cannot be scanned beyond the straight position in the horizontal direction, except by adjustment of the support table.

Standard equations are used to convert between the {omega, kappa, phi} setting and the more commonly used {theta, chi, phi} notation. These conversions will be made in user software, so it is not necessary for this to be a function of the diffractometer control hardware. Because scans of the values of the converted angles will be made, it is necessary that simultaneous motions be possible on multiple axes.

In the layout shown in the figure, the detector motions are shown on the *outside* of the main vertical stand. This is to facilitate and conserve the mutual alignment of the omega and omega_D axes, since these have to be the most precise of all combinations. The reversed stacking also allows the detector arm to be supported as far away from the instrument as possible, which will minimize its gravitational deflection. This will also



reduce the size of counterweights associated with the detector arm.

All of the sample axes must be suitably counterbalanced so that large static torques are avoided on their drive mechanisms. This is not required for the temporary loading of the sample axes during installation of the vacuum system. The counterweights of the detector arms should be coarsely adjustable by adding and subtracting weights or by moving them. The maximum detector-arm load is an X95 rail plus a 10kg detector located up to 3m from the center point.